Pace Analytical Services, Inc.
315 Chestnut Street
Virginia, MN 55792
(218) 742-1042

July 18, 2016

Tom Moe
USS Corporation
P.O. Box 417
8771 Park Ridge Dr
Mountain Iron, MN 55768

RE: Project: NPDES-TB WK1
Pace Project No.: 1269769

Dear Tom Moe:
Enclosed are the analytical results for sample(s) received by the laboratory on July 06, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Melisa M Woods
melisa.woods@pacelabs.com
Project Manager

Enclosures

cc: Cory Hertling
Terri Sabetti, NTS

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CERTIFICATIONS

Project: NPDES-TB WK1
Pace Project No.: 1269769

Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792
Alaska Certification #MN01084
Arizona Department of Health Certification #AZ0785
Minnesota Dept of Health Certification #: 027-137-445
North Dakota Certification: # R-203
Wisconsin DNR Certification # : 998027470
WA Department of Ecology Lab ID# C1007
Nevada DNR #MN010842015-1
Oklahoma Department of Environmental Quality

Duluth Minnesota Cerification ID's

4730 Oneota St., Duluth, MN 55807
Minnesota Dept of Health Certification #: 027-137-152
Wisconsin DNR Certification # : 999446800
North Dakota Certification #: R-105

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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SAMPLE SUMMARY

Project: NPDES-TB WK1
Pace Project No.: 1269769

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1269769001	**SD 001 (Seep 020)**	Water	07/06/16 11:20	07/06/16 13:25

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SAMPLE ANALYTE COUNT

Project: NPDES-TB WK1
Pace Project No.: 1269769

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1269769001	**SD 001 (Seep 020)**	EPA 1664 TPH	BT1	1	PASI-DUL
		USGS I-3765	BEM	1	PASI-V
		EPA 300.0	CSD	1	PASI-V

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ANALYTICAL RESULTS

Project: NPDES-TB WK1
Pace Project No.: 1269769

Sample: SD 001 (Seep 020) **Lab ID: 1269769001** Collected: 07/06/16 11:20 Received: 07/06/16 13:25 Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
1664 SGT-HEM, TPH	Analytical Method: EPA 1664 TPH								
Total Petroleum Hydrocarbons	ND	mg/L	3.0	1.0	1		07/11/16 16:44		
USGS I-3765 TSS	Analytical Method: USGS I-3765								
Total Suspended Solids	**6.4**	mg/L	1.0	1.0	1		07/12/16 12:08		
300.0 IC Anions 28 Days	Analytical Method: EPA 300.0								
Sulfate	**1010**	mg/L	20.0	10.0	10		07/13/16 00:24	14808-79-8	

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB WK1
Pace Project No.: 1269769

QC Batch: 87251 Analysis Method: EPA 1664 TPH
QC Batch Method: EPA 1664 TPH Analysis Description: 1664 SGT-HEM, TPH
Associated Lab Samples: 1269769001

METHOD BLANK: 341660 Matrix: Water
Associated Lab Samples: 1269769001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Petroleum Hydrocarbons	mg/L	ND	3.0	1.0	07/11/16 14:30	

LABORATORY CONTROL SAMPLE: 341661

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	20	16.3	82	64-132	

MATRIX SPIKE SAMPLE: 341662

Parameter	Units	1269658001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	<1.1	20.2	13.2	65	64-132	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB WK1
Pace Project No.: 1269769

QC Batch: 87443 Analysis Method: USGS I-3765
QC Batch Method: USGS I-3765 Analysis Description: USGS I-3765 Total Suspended Solids
Associated Lab Samples: 1269769001

METHOD BLANK: 342377 Matrix: Water
Associated Lab Samples: 1269769001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Suspended Solids	mg/L	ND	1.0	1.0	07/12/16 12:07	

LABORATORY CONTROL SAMPLE: 342378

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Suspended Solids	mg/L	239	230	96	80-120	

SAMPLE DUPLICATE: 342379

Parameter	Units	1269979001 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	154	160	4	10	

SAMPLE DUPLICATE: 342380

Parameter	Units	1269981002 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	28.0	26.0	7	10	

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REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

Project: NPDES-TB WK1
Pace Project No.: 1269769

QC Batch: 87475 Analysis Method: EPA 300.0
QC Batch Method: EPA 300.0 Analysis Description: 300.0 IC Anions
Associated Lab Samples: 1269769001

METHOD BLANK: 342589 Matrix: Water
Associated Lab Samples: 1269769001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Sulfate	mg/L	ND	2.0	1.0	07/13/16 00:03	

LABORATORY CONTROL SAMPLE: 342590

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Sulfate	mg/L	50	48.9	98	90-110	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 342591 342592

Parameter	Units	1269769001 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	1010	500	500	1510	1510	100	100	90-110	0	20	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 342593 342594

Parameter	Units	1270031004 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Sulfate	mg/L	17.4	50	50	69.3	70.1	104	105	90-110	1	20	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

REPORT OF LABORATORY ANALYSIS

Pace Analytical Services, Inc.
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QUALIFIERS

Project: NPDES-TB WK1
Pace Project No.: 1269769

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-DUL	Pace Analytical Services - Duluth
PASI-V	Pace Analytical Services - Virginia

Pace Analytical Services, Inc.
315 Chestnut Street
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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES-TB WK1
Pace Project No.: 1269769

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1269769001	**SD 001 (Seep 020)**	EPA 1664 TPH	87251		
1269769001	**SD 001 (Seep 020)**	USGS I-3765	87443		
1269769001	**SD 001 (Seep 020)**	EPA 300.0	87475		

PaceAnalytical

CHAIN-OF-CUSTODY / Analytical Request Docu

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be

WO#: 1269769
PM: MMW Due Date: 07/20/16
CLIENT: USS CORP

Page : 1 of

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:
Company: USS Corporation	Report To: Tom Moe	Attention:
Address: P.O. Box 417	Copy To:	Company Name:
Mt. Iron, MN 55768		Address:
Email:	Purchase Order #:	Pace Quote:
Phone: Fax:	Project Name: NPDES-TB Wk1	Pace Project Manager: heather.zika@pacelabs.com,
Requested Due Date:	Project #:	Pace Profile #:

MATRIX / CODE: Drinking Water DW; Water WT; Waste Water WW; Product P; Soil/Solid SL; Oil OL; Wipe WP; Air AR; Other OT; Tissue TS

ITEM #	SAMPLE ID (One Character per box. (A-Z, 0-9 / , -) Sample Ids must be unique)	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED START DATE	START TIME	END DATE	END TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Unpreserved	H2SO4	HNO3	HCl	NaOH	Na2S2O3	Methanol	Other	Analyses Test Y/N	TSS,SO4	TRPH 1664	Residual Chlorine (Y/N)
1	SD 001 (Seep 020)	WT		7-6-16	11:20	7-6-16	11:20												x	x	

Items 2–12: blank

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	TEMP in C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
	[illegible]	7-6-16	13:25	[illegible]	7/6	1335	4.7	Y	N	Y

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: [illegible]

SIGNATURE of SAMPLER: [illegible] DATE Signed: 7-6-16

Pace Analytical	Document Name: Sample Condition Upon Receipt Form	Document Revised: 23Feb2015 Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: USS Corporation

Project #: WO# : 1269769

1269769

Courier: ☐Fed Ex ☐UPS ☐USPS ☒Client ☐Commercial ☐Pace ☐Other:

Tracking Number:

Custody Seal on Cooler/Box Present? ☐Yes ☒No **Seals Intact?** ☐Yes ☒No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐Bubble Wrap ☐Bubble Bags ☒None ☐Other: **Temp Blank?** ☒Yes ☐No

Thermometer Used: ☒ 140792808 **Type of Ice:** ☒Wet ☐Blue ☐None ☐Samples on ice, cooling process has begun

Cooler Temp Read °C: 4.4 **Cooler Temp Corrected °C:** 4.7 **Biological Tissue Frozen?** ☐Yes ☐No ☒NA

Temp should be above freezing to 6°C Correction Factor: +0.3 **Date and Initials of Person Examining Contents:** AT 7/06/16

Comments:

Chain of Custody Present?	☒Yes	☐No	☐N/A	1.
Chain of Custody Filled Out?	☒Yes	☐No	☐N/A	2.
Chain of Custody Relinquished?	☒Yes	☐No	☐N/A	3.
Sampler Name and Signature on COC?	☒Yes	☐No	☐N/A	4.
Samples Arrived within Hold Time?	☒Yes	☐No	☐N/A	5.
Short Hold Time Analysis (<72 hr)?	☐Yes	☒No	☐N/A	6.
Rush Turn Around Time Requested?	☐Yes	☒No	☐N/A	7.
Sufficient Volume?	☒Yes	☐No	☐N/A	8.
Correct Containers Used?	☒Yes	☐No	☐N/A	9.
-Pace Containers Used?	☒Yes	☐No	☐N/A	
Containers Intact?	☒Yes	☐No	☐N/A	10.
Filtered Volume Received for Dissolved Tests?	☐Yes	☐No	☒N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: W1	☒Yes	☐No	☐N/A	12.
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐Yes	☐No	☒N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐Yes	☐No	☒N/A	13.
Headspace in VOA Vials (>6mm)?	☐Yes	☐No	☒N/A	14.
Trip Blank Present?	☐Yes	☐No	☒N/A	15.
Trip Blank Custody Seals Present?	☐Yes	☐No	☒N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐Yes ☐No

Person Contacted: Date/Time:

Comments/Resolution:

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: M Lisa Woods **Date:** 7/7/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

Intra-Regional Chain of Custody

Pace Analytical®
www.pacelabs.com

Workorder: 1269769 **Workorder Name:** NPDES-TB WK1 **Owner Received Date:** 7/6/2016 **Due Date:** 7/20/2016

Received at:	Send To Lab:
Pace Analytical Virginia 315 Chestnut Street Virginia, MN 55792 Phone (218) 742-1042	Pace Analytical Duluth 4730 Oneota Street Duluth, MN 55807 Phone (218) 727-6380

Report To:
Melisa M Woods

Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	Preserved Containers: HCL	Requested Analysis: EPA 1664 TPH	LAB USE ONLY
1	SD 001 (Seep 020)	PS	7/6/2016 11:20	1269769001	Water	1	X	
2								
3								
4								
5								

Transfers	Released By	Date/Time	Received By	Date/Time
1		7/7/16 1400		7/7/16 1515
2				
3				
4				

Comments

Cooler Temperature on Receipt 1.2 °C **Custody Seal** Y or N **Received on Ice** Y or N **Samples Intact** Y or N

****In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document. This chain of custody is considered complete as is since this information is available in the owner laboratory.*

Pace Analytical®	Document Name: Sample Condition Upon Receipt Form	Document Revised: 22Jan2016 Page 1 of 1
	Document No.: F-DUL-C-001-Rev.01	Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition Upon Receipt

Client Name: IR COC

Project #:

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace ☒ Other: Jake

Tracking Number:

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No **Seals Intact?** ☒ Yes ☐ No

Optional: Proj. Due Date: Proj. Name:

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: **Temp Blank?** ☒ Yes ☐ No

Thermometer Used: ☒ B00051 **Type of Ice:** ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 2.0 **Cooler Temp Corrected °C:** 1.2 **Biological Tissue Frozen?** ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C Correction Factor: -0.8 °C **Date and Initials of Person Examining Contents:** RE 7/8/16

Comments:

Chain of Custody Present?	☒ Yes	☐ No	☐ N/A	1.
Chain of Custody Filled Out?	☒ Yes	☐ No	☐ N/A	2.
Chain of Custody Relinquished?	☒ Yes	☐ No	☐ N/A	3.
Sampler Name and Signature on COC?	☒ Yes AP 7-8-16	☐ No	☒ N/A	4.
Samples Arrived within Hold Time?	☒ Yes	☐ No	☐ N/A	5.
Short Hold Time Analysis (<72 hr)?	☐ Yes	☒ No	☐ N/A	6.
Rush Turn Around Time Requested?	☐ Yes	☒ No	☐ N/A	7.
Sufficient Volume?	☒ Yes	☐ No	☐ N/A	8.
Correct Containers Used?	☒ Yes	☐ No	☐ N/A	9.
-Pace Containers Used?	☒ Yes	☐ No	☐ N/A	
Containers Intact?	☒ Yes	☐ No	☐ N/A	10.
Filtered Volume Received for Dissolved Tests?	☐ Yes	☐ No	☒ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC? -Includes Date/Time/ID/Analysis Matrix: WT	☒ Yes	☐ No	☐ N/A	12.
All containers needing acid/base preservation will be checked and documented in the pH logbook.	☐ Yes	☐ No	☒ N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	☐ Yes	☐ No	☐ N/A	13.
Headspace in VOA Vials (>6mm)?	☐ Yes	☐ No	☐ N/A	14.
Trip Blank Present?	☐ Yes	☐ No	☐ N/A	15.
Trip Blank Custody Seals Present?	☐ Yes	☐ No	☐ N/A	
Pace Trip Blank Lot # (if purchased):				

CLIENT NOTIFICATION/RESOLUTION **Field Data Required?** ☐ Yes ☐ No

Person Contacted: Date/Time:

Comments/Resolution:

FECAL WAIVER ON FILE Y N TEMPERATURE WAIVER ON FILE Y N

Project Manager Review: AP for LMF **Date:** 7-8-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)